

### INJECTION MOULDED CONTAINERS

The present invention relates to a process for preparing a container by injection moulding a composition comprising a water soluble polymer, such as poly(vinyl alcohol) (PVOH).

Clothes washing compositions may be delivered to a clothes washing machine by a delivery tray from which the composition is fed into the washing drum, or they may be placed directly into the washing drum. The washing compositions may be in powder, liquid or block form. Liquid compositions have the disadvantage that they may be spilt. The same applies to powder compositions. Powder compositions have the additional disadvantage that they may produce dust which can be inhaled. These problems are overcome or lessened when blocks of washing composition are used. These are normally individually wrapped. On unwrapping a block, for use, it is still possible that some dust may be produced. Additionally it is an inconvenience for the consumer to have to unwrap the block. Furthermore it is almost impossible for the user to avoid some contact between the block and his or her skin, so leading to a requirement for the user to wash their hands after starting the washing machine. In fact, all of the methods described involve a risk of contact between the composition and the skin, and it is desirable in all cases for the user to wash their hands after starting the washing machine. In this context it should be borne in mind that many compositions contain enzymes to assist the cleaning action. Even though the user may tolerate enzyme residues which may be left in clothes after washing,

they may still not tolerate contact between the concentrated washing composition containing the enzymes, and the skin.

5 Similar considerations apply in relation to other areas including fabric care, surface care and dishwashing. Thus, in relation in particular to dishwashing compositions, there are also problems of spillage, dust generation, skin contact and inconvenience.

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It is known to package chemical compositions which may be of a hazardous or irritant nature in water-soluble or water-dispersible materials such as films. The package can simply be added to water in order to dissolve or disperse  
15 the contents of the package into the water.

For example, WO 89/12587 discloses a package which comprises an envelope of a water-soluble or water-dispersible material which comprises a flexible wall and  
20 a water-soluble or water-dispersible heat seal. The package may contain an organic liquid comprising, for example, a pesticide, fungicide, insecticide or herbicide.

CA-A-1,112,534 discloses a packet made of a water-soluble  
25 material in film form enclosing within it a paste-form, automatic dishwasher-compatible detergent composition. The water-soluble material may be, for example, poly(vinyl alcohol), polyethylene oxide or methyl cellulose.

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It is also known to form water-soluble containers by thermoforming a water-soluble material. For example, WO

92/17382 discloses a package containing an agrochemical such as a pesticide comprising a first sheet of non-planar water-soluble or water-dispersible material and a second sheet of water-soluble or water-dispersible material superposed on the first sheet and sealed to it by a continuous closed water-soluble or water-dispersible seal along a continuous region of the superposed sheets.

The above methods of packaging have, however, a number of disadvantages.

The first disadvantage is that they do not have a particularly attractive appearance. In fields such as containers used in the domestic environment, an attractive appearance for an article is extremely desirable. Liquids contained in envelopes of water-soluble film can have a limp, unattractive appearance.

The second disadvantage is that it is difficult to form two or more separate compartments in the packaging so that two incompatible components are both enclosed but separated from each other. Although an arrangement has been described to separate incompatible materials in flexible pouches in WO 93/08095, the method proposed is complex and is not currently achievable in large-scale manufacturing. It cannot, therefore, be used for producing large numbers of containers.

The third disadvantage is that there is only limited control of the release profile of the compositions held in the containers. For example, when a composition is held between two planar water-soluble films or in a thermo-

formed package, the composition is simply released at the time when the films dissolve or disperse in water. While it may be possible to control to a certain extent the timing of the start of release of the contents, there can  
5 be no control over the rate of release of the contents since the entire film dissolves or disperses at about the same time. Furthermore it can be difficult to provide an extended time before the contents of the package are released. An additional problem also arises with thermo-  
10 formed packages. If the thermoforming is not carefully controlled there may be inadvertent thinning of the film material at the points where the material is drawn down into the mould when it is thermoformed. This could release the contents of the package early. Additionally,  
15 in all of the above packages, it is not possible to release different compositions at different times or at different rates since, as discussed above, it is not possible to incorporate more than one composition in each water-soluble container.

20

The fourth disadvantage is that the containers cannot be produced at a particularly fast rate. When the containers are produced by heat-sealing planar films or by thermoforming, the containers have to be immediately filled  
25 and sealed. All of these procedures have to be carried out in succession. This means that it is not possible to obtain a quick throughput for mass-market goods such as household products. For example, standard thermoforming machines can only produce around 400 to 800 containers  
30 per minute.

The present invention seeks to provide water-soluble containers which overcome some or all of the above disadvantages.

5 The present invention provides a process for the manufacture of a single or multi-compartment, rigid, water-soluble container, containing a detergent composition, wherein the container is at least partially formed of injection moulded water soluble polymer; the process comprising the steps of forming the container, filling with  
10 the detergent composition and sealing, wherein the container is allowed to contact / brought into contact with a plasticiser after sealing.

15 Preferably the plasticiser is gaseous or in vapour form.

Generally the plasticiser is water. Thus the present invention provides a process for the manufacture of a single or multi-compartment, rigid, water-soluble container,  
20 containing a detergent composition, wherein the container is at least partially formed of injection moulded water soluble polymer; the process comprising the steps of forming the container, keeping the container under substantially anhydrous conditions, filling with the detergent composition and sealing, wherein the container is  
25 allowed to contact / brought into contact with a plasticiser after sealing.

Surprisingly we have found that when the process of the  
30 present invention is used a rigid water soluble container is produced. More specifically by keeping the injection moulded container in substantially anhydrous conditions

after formation and before filling with the detergent composition the rigidity of the container is preserved.

5 In this way we have found that in the preferred sealing process (see later) wherein a top sealing film is applied (usually in a pressure contact process) the container exhibits sufficient rigidity for effective joining/sealing of the film to occur.

10 Additionally we have also found that during the preferred filling operation (see later) the high rigidity of the container is especially beneficial.

15 The filling operation is usually performed using a dispensing apparatus. The dispensing apparatus commonly comprises a nozzle which directs the composition to be filled into the container. For multi-compartment containers, especially where each compartment is filled with a separate composition, it is vital that each compartment  
20 be arranged relative to its appropriate dispensing means with high predictability to avoid incorrect dispense. This would otherwise lead to detrimental interaction of compartment compositions (if more than one composition is dispensed into one compartment) or wastage of compartment  
25 compositions (if a composition is dispensed outside the container). The method in accordance with the invention allows this high level of predictability to be achieved. Therefore filling issues caused by incorrect dispense are minimised.

30

The anhydrous retention conditions may be effected using common environmental controlled means. As an example, if

the containers are to be stored before filling the storage conditions need to be controlled so that the humidity level is low. This can be achieved by the use of dehumidifiers controlling the atmosphere of the area where  
5 the containers are stored. Alternatively a number of containers may be stored in a sealed enclosure (such as a water-tight bag/box, e.g. a metal/plastic vessel) from which the bulk of the available moisture is withdrawn. The latter method is particularly suitable, where the  
10 containers need to be transported from the site of formation to the site of filling.

Most preferably the containers are kept under substantially anhydrous conditions until filling.

15 The containers produced in accordance with the invention need not be kept under anhydrous conditions (once filled with the detergent composition and sealed). After time the containers will begin to absorb moisture either from  
20 the air or from the composition held within (if the composition is an aqueous liquid composition containing free water). It is believed that the water plasticises the water-soluble polymer, which was dried during the injection moulding process. It is believed that water (from  
25 the air or from an aqueous composition held within the container) lowers the rigidity of the container.

The reduction in rigidity has several benefits. Firstly, although high rigidity (as described above) is beneficial  
30 during the manufacturing (filling and/or sealing) process, consumers generally find overly rigid containers unappealing. Following moisture uptake the container de-

velops a desirable tactile quality highly regarded by consumers.

Additionally the reduction in rigidity (after processing)  
5 improves container stability in storage/transport. The low rigidity containers display a much greater ability to withstand impact during storage and transport. Thus undesirable rupture of containers is vastly reduced.

10 The containers have the usual advantages associated with injection moulded containers. The containers have an attractive, uniform appearance which does not vary between different containers. Also, a wide variety of different shapes and designs are available. Furthermore, rigid  
15 containers can easily have various elements incorporated which are considered to be pleasing to the eye.

Moreover, it is possible to control the release profile of the contents of the container. Since the container is  
20 rigid, it is possible to adapt the width of all of the walls of the container to control both the start of release of the composition as well as the rate of release. For example, one or more walls may be made thin in order to have an early release of the composition. Alternately  
25 all the walls may be thick in order to ensure that there is a delayed release of the composition. The rate of release of the composition may also be controlled by ensuring that only part of the container has thin walls which are dissolved or dispersed before the remainder of  
30 the container.



Preferably the water soluble polymer is PVOH or a derivative thereof.

Other water-soluble polymers may be used either as an alternative or in addition to PVOH. Preferred examples include poly(vinylpyrrolidone), poly(acrylic acid), poly(maleic acid), a cellulose derivative (such as an ether or hydroxypropyl methyl cellulose); and poly(glycolide), poly(glycolic acid), poly(lactides),  
10 poly (lactic acid) or a copolymer thereof

It will be appreciated that the container may be made in part by any other water-soluble polymer. Therefore, a further feature of the invention is a rigid, water-soluble container made of at least two injection moulded polymers, a "first" water-soluble polymer, preferably selected from poly(vinyl alcohol); a cellulose derivative (such as an ether or hydroxypropyl methyl cellulose); and poly(glycolide), poly(glycolic acid), poly(lactides),  
15 poly (lactic acid) or a copolymer thereof; and a "second" water-soluble polymer which polymer when dissolved in water is active in detergency.

Thus in an alternative release control mechanism different walls or parts of walls of the container may be prepared from different water-soluble polymers which have different dissolution characteristics. For example, a first compartment may be fully enclosed by a polymer which dissolves at a higher or lower temperature than the polymer enclosing a second compartment. Thus different  
25 components can be released at different times. If the container holds a solid or gelled composition, it is not  
30

even necessary for the container to fully enclose the composition. A part may be left exposed, so that it immediately begins to dissolve when added to water.

5 It is possible for suitable additives such as plasticizers (other than water) and lubricants to be included. Plasticizers are generally used in an amount of up to 20wt%, for example from 8 to 20wt%, lubricants are generally used in an amount of 0.5 to 5wt% and the polymer is  
10 generally therefore used in an amount of 75 to 84.5wt%, based on the total amount of the moulding composition.

Surprisingly we have found that if a plasticiser having a melting higher than room temperature is used then the rigidity of the containers formed is extremely high. This  
15 has the advantage that the containers maintain their shape after injection moulding. This in particular surprising as water-soluble polymers, especially PVOH, have the non-desired characteristic to easily deform after the  
20 shaping process.

Thus preferred plasticisers comprise a carbohydrate. Carbohydrates are usually represented by the generalised formula  $C_x(H_2O)_y$ . The term herein also includes materials  
25 which are similar in nature like gluconic acids or amino sugars which cannot be fully represented by this formula. Other carbohydrate derivatives like sugar alcohols such as sorbitol, glucitol, mannitol, galactitol, dulcitol, xylitol, erythritol, isomaltutose and isomalt fall within  
30 this term.

Most preferred carbohydrates include the more thermally

stable carbohydrates such as sorbitol, glucitol, mannitol, galactitol, dulcitol, xylitol, erythritol, isomaltulose and isomalt.

- 5 We have also found that when the containers are formed in the substantial absence of a plasticiser which is liquid at room temperature then the rigidity of the containers after shaping is extremely high.
- 10 The container is generally cold water (20°C) soluble, but may be insoluble in cold water at 20°C and only become soluble in warm water or hot water having a temperature of, for example, 30°C, 40°C, 50°C or even 60°C.
- 15 For certain applications or uses, containers soluble in aqueous environments at temperatures as low as 5°C are also desirable.

In order to ensure that the polymer is capable of being  
20 injection moulded, it is usual to incorporate components such as plasticizers (as discussed above) and mould release agents in an amount of up to, for example, 15wt% of the composition. Solids such as talc, stearic acid, magnesium stearate, silicon dioxide, zinc stearate, and col-  
25 loidal silica may be used as mould release agents.

Poly(vinylpyrrolidone) may be moulded at temperatures of from 120-180°C, depending upon the formulation selected and the melt flow index required.

Poly(acrylic acid) may be moulded at temperatures of from 180-220°C, for example, depending upon the formulation selected and the melt flow index required.

- 5 Poly(maleic acid) may be moulded at temperatures of, from 180-220°C for example, depending upon the formulation selected and the melt flow index required.

- PVOH may be moulded at temperatures of, for example, from  
10 180-220°C, depending upon the formulation selected and the melt flow index required. The PVOH preferably used to form the container of the present invention may be partially / fully alcoholised or hydrolysed. For example it may be from 40-99%, preferably 70-92%, more preferably  
15 about 88%, alcoholised or hydrolysed polyvinylacetate.

Preferably the container is a container enclosing a washing composition.

- 20 All of the polymer compositions may also include other components such as colouring agents and components which modify their properties.

- Injection moulding techniques are well known to the  
25 skilled person and are well described in the literature (see, for example a good summary is provided in "The Wiley Encyclopedia of Packaging Technology" Wiley Interscience 1986). Special techniques, described below, are preferred features of the invention for producing con-  
30 tainers having more than one type of polymer.

Simultaneous injection moulding

1) two or more polymers are molten mixed and injected  
into a mould;

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2) two or more polymers are injected into a mould through  
more than one gate, each gate allowing simultaneous  
injection of a single polymer or molten mix into the  
mould;

10

3) simultaneously injection moulding two or more compart-  
ments and then joining the compartments together.

Sequential injection moulding

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1) multi-component injection moulding;

2) sandwich injection moulding;

20 3) sequentially injection moulding two or more;

4) compartments and then joining the compartments to-  
gether.

25 Multi-component injection moulding covers two distinct  
processes

A) injection moulding a polymer or molten polymer  
mix into a mould, removing the solid polymer and  
30 inserting it into a second mould and injection  
moulding a second polymer or polymer mix into  
the second mould;

B) injection moulding a polymer or molten polymer mix into a part of a mould, injection moulding a second polymer or molten polymer mix into a further part of the mould.

Steps A) and B) may be repeated more than once and may be combined. It will be appreciated by the skilled person that the first injection moulded polymer must survive the pressure and temperature conditions of the second, or subsequent, injection moulding.

For step B) the first polymer or molten mix may be prevented from entering parts of the mould by any physical means, such as, gates, gravity, positive or negative pressure.

Sandwich injection moulding (or sometimes called skin-core injection moulding) comprises injection moulding a polymer or molten polymer mix into a mould until it is partially filled and then injecting a second polymer or molten polymer mix into the same mould through the same gate to form the core. An additional step of sealing the core may be performed.

It will be appreciated that any combination of simultaneous and sequential injection moulding may be used.

The closure part may itself be injection moulded or blow moulded. Preferably, however, it is a plastics film secured over the receptacle part. The film may, for exam-

ple, comprise PVOH or a cellulose ether such as HPMC or another water-soluble polymer.

Preferably for closing the container the receptacle part  
5 and the closure are sealed together, for example by heat  
sealing. A suitable heat sealing temperature is 120 to  
195°C, more preferably 140 to 150°C. The sealing pres-  
sure depends on the heat sealing machine used. A suit-  
able sealing pressure is from 250 to 800 kPa (35 to 120  
10 p.s.i.). Examples of sealing pressures are 400 to 800  
kPa (60 to 120 p.s.i.), especially 276 to 552 kPa (40 to  
80 p.s.i.), more especially 345 to 483 kPa (50 to 70  
p.s.i.). Suitable sealing dwell times are at least 0.4  
seconds, for example 0.4 to 2.5 seconds.

15 Other methods of sealing the receptacle part to the clo-  
sure together may be used. These include infra-red, ra-  
dio frequency, ultrasonic, laser, solvent, vibration,  
electromagnetic, hot gas, hot plate, insert bonding,  
20 fraction sealing or spin welding. An adhesive such as  
water or an aqueous solution of PVOH may also be used.  
The adhesive can be applied to the sheets by spraying,  
transfer coating, roller coating or otherwise coating, or  
the sheets can be passed through a mist of the adhesive.

25 The container walls have thicknesses such that the con-  
tainers are rigid (as described above). For example, the  
outside walls and any inside walls which have been injec-  
tion moulded independently have a thickness of greater  
30 than 100µm, for example greater than 150µm or greater  
than 200µm, 300µm, or 500µm, 750µm or 1mm. Generally the  
walls are moulded so as to be as thin as possible to re-

duce container material consumption in the process and to make the dissolution time of the container as short as possible. Usually the wall thickness at more than 60% of the wall area is less than 800 $\mu$ m, more preferably less than 600 $\mu$ m and most preferably less than 400 $\mu$ m. Preferably, the closure part is of a thinner material than the receptacle part. If different compartments having different dissolution times are required, different wall thicknesses can be used. A thickness difference of from 100 $\mu$ m to 500 $\mu$ m, preferably from 250 $\mu$ m to 350m, would give a suitable difference in release times.

Preferably, the closure part dissolves in water (at least to the extent of allowing the washing composition in the receptacle part to be dissolved by the water; and preferably completely) at 40°C in less than 5 minutes, preferably in less than 2 minutes.

The receptacle part and the closure part could be of the same thickness or different thicknesses. The closure part may, for example, be of higher solubility than the receptacle part, in order to dissolve more quickly.

Preferably, the washing container is generally cuboid in its external shape, with the top wall being formed by the closure part, and with the side walls and base wall being formed by the receptacle part.

Preferably, a washing container of the invention is manufactured by forming an array of receptacle parts, each receptacle part being joined to adjacent receptacle parts, and being separable from them by a snap or tear



action. The array is preferably one which has columns and rows of the receptacle parts. The receptacle parts may be separated by frangible webs of the water-soluble polymer such as PVOH or a cellulose ether.

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Alternatively, the receptacle parts may be manufactured with the aforementioned flanges, such that they are separated from each other by a line of weakness. For example the material may be thinner, and so able to be broken or  
10 torn readily. The thinness may be a result of the moulding process or, preferably, of a later scoring step.

In the manufacturing method, the array, formed by injection moulding, is fed to a filling zone, and all the receptacle parts are charged with the washing composition.  
15 A sheet of a water-soluble polymer such as PVOH or a cellulose ether may then be secured over the top of the array, to form the closure parts for all the receptacle parts of the array. The array may then be split up into  
20 the individual washing capsules, prior to packaging, or it may be left as an array, for packaging, to be split by the user.

The container, capsule or receptacle part defines two or  
25 more compartments, which may contain different products useful in a washing process. In such a situation a dividing wall or walls of the compartments preferably terminate at the top of the container, i.e. in the same plane as the top edges of the side walls, so that when  
30 the receptacle part is closed by the closure part the contents of the compartments cannot mix. The container may be provided with an upstand, preferably spaced from

the side walls thereof, and preferably of generally cylindrical shape. If wished, the remaining volume of the container can be divided into two or more parts by means of walls extending between the upstand and the side walls.

The container may be formed with an opening, for example a depression, formed in the side wall or the base wall, and preferably being open in the outward direction. That is to say, it preferably does not form part of the main volume defined by the container. Preferably the opening is adapted to receive, in a press-fit manner, a solid block (for example a tablet) of a composition, for example a material useful in a washing process.

Preferably, the closure part is of a transparent or translucent material, so that the contents of the washing capsule can be seen.

Preferably, the container is of a transparent or translucent material, so that the contents of the washing capsule can be seen.

The washing composition within the container, or within a compartment thereof, need not be uniform. For example during manufacture it could be fed first with a settable agent, for example a gel, useful in a washing process, and then with a different material. The first material could dissolve slowly in the washing process so as to deliver its charge over a long period within the washing process. This might be useful, for example, to provide

immediate, delayed or sustained delivery of a softening agent in a clothes washing container.

The container may, for example, be in at least two parts  
5 (a body part and a cap part) which fit tightly, and preferably sealingly and inseparably, together to form a compartment in which is stored the ingredient to be achieved. In one example, the container or capsule may have three parts - a body such as a receptacle part, a  
10 first cap, and then a second cap to fit over the closed end of either the body or the first cap, so as to result in a capsule with two separate compartments. Where there are three such parts (or more; four parts - a body and three caps - make three compartments, and so on), then  
15 naturally the ingredients in each compartment may be the same or they may be different.

In all embodiments of the present invention one compartment may contain, for example, a liquid or solid component  
20 (such as a powder, granules or a compressed or gelled tablet) and another may contain a different liquid or solid component (such as a powder, granules or a compressed or gelled tablet). Alternatively, more than one component may be present in one or more compartments.  
25 For example a compartment may contain a solid component, for example in the form of a ball or pill (such as a powder, granules or a compressed or gelled tablet), and a liquid component.

30 Desirably the composition has a mass of at least 10g or 15g, for example, from 10g or 15g to 100g, especially from 10g to 15g to 40g. For example, a dishwashing com-

position may weigh from 10g or 15g to 20g, a water-softening composition may weigh from 25g to 35g, and a laundry composition may weigh from 10g to 40g, 20g to 40g or 30g to 40g.

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In general the maximum dimension of the container is 5cm. For example, a cuboid container may have a length of 1 to 5cm, especially 3.5 to 4.5cm, a width of 1.5 to 3.5cm, especially 2 to 3cm, and a height of 1 to 2cm, especially  
10 1.25 to 1.75cm.

The composition may comprise a powder, gel, paste or low water liquid foundation.

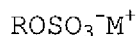
15 The composition contained by the container may be, for example, any which is suitable for the designated application, for example a clothes washing or dishwashing application. It may be a powder or a liquid but if a liquid, may be a low water formulation, preferably having a  
20 maximum water content of 5wt%, in order to maintain the integrity of the walls of the capsule or a higher water formulation containing, for example, at least 8wt% water. It will be appreciated that higher water contents may be present where the water is chemically or physically  
25 bound. The composition may be formulated having regard to the fact that the user will not come into contact with the composition, whether by inhalation or by skin contact. For example, the composition may include an enzyme, without concern about physical contact between the  
30 composition containing the enzyme, and the user.

If the container contains an aqueous liquid having a relatively high free water content, it may be necessary to take steps to ensure the liquid does not attack the water-soluble polymer if it is soluble in cold water (20°C), or water at a temperature of up to, say, 35°C. Steps may be taken to treat the inside surfaces of the container, for example by coating it with agents such as PVC (poly (vinylidene chloride)) or PTFE (polytetrafluoroethylene), or to adapt the composition to ensure that it does not dissolve the polymer. For example, it has been found that ensuring the composition has a high ionic strength or contains an agent which minimises water loss through the walls of the container will prevent the composition from dissolving the polymer from the inside. This is described in more detail in EP-A-518,689 and WO 97/27743.

The composition held within the container depends, of course, on the intended use of the composition. It may, for example, contain surface active agents such as an anionic, non-ionic, cationic, amphoteric or zwitterionic surface active agent or mixture thereof.

Examples of anionic surfactants are straight-chained or branched alkyl sulfates and alkyl polyalkoxylated sulfates, also known as alkyl ether sulfates. Such surfactants may be produced by the sulfation of higher C<sub>8</sub>-C<sub>20</sub> fatty alcohols.

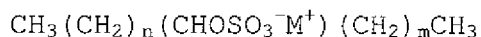
Examples of primary alkyl sulfate surfactants are those of formula:



wherein R is a linear C<sub>8</sub>-C<sub>20</sub> hydrocarbyl group and M is a water-solubilising cation. Preferably R is C<sub>10</sub>-C<sub>16</sub> alkyl, for example C<sub>12</sub>-C<sub>14</sub>, and M is alkali metal such as lithium, sodium or potassium.

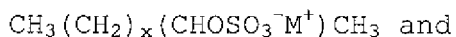
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Examples of secondary alkyl sulfate surfactants are those which have the sulfate moiety on a "backbone" of the molecule, for example those of formula:

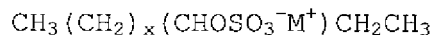


10 wherein m and n are independently 2 or more, the sum of m+n typically being 6 to 20, for example 9 to 15, and M is a water-solubilising cation such as lithium, sodium or potassium.

15 Especially preferred secondary alkyl sulfates are the (2,3) alkyl sulfate surfactants of formulae:



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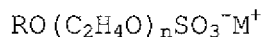


for the 2-sulfate and 3-sulfate, respectively. In these formulae x is at least 4, for example 6 to 20, preferably 10 to 16. M is cation, such as an alkali metal, for example lithium, sodium or potassium.

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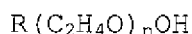
Examples of alkoxyated alkyl sulfates are ethoxylated alkyl sulfates of the formula:

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wherein R is a C<sub>8</sub>-C<sub>20</sub> alkyl group, preferably C<sub>10</sub>-C<sub>18</sub> such as a C<sub>12</sub>-C<sub>16</sub>, n is at least 1, for example from 1 to 20, preferably 1 to 15, especially 1 to 6, and M is a salt-forming cation such as lithium, sodium, potassium, ammonium, alkylammonium or alkanolammonium. These compounds can provide especially desirable fabric cleaning performance benefits when used in combination with alkyl sulfates.

- 10 The alkyl sulfates and alkyl ether sulfates will generally be used in the form of mixtures comprising varying alkyl chain lengths and, if present, varying degrees of alkoxylation.
- 15 Other anionic surfactants which may be employed are salts of fatty acids, for example C<sub>8</sub>-C<sub>18</sub> fatty acids, especially the sodium or potassium salts, and alkyl, for example C<sub>8</sub>-C<sub>18</sub>, benzene sulfonates.
- 20 Examples of nonionic surfactants are fatty acid alkoxy-  
lates, such as fatty acid ethoxylates, especially those of formula:



25

wherein R is a straight or branched C<sub>8</sub>-C<sub>16</sub> alkyl group, preferably a C<sub>9</sub>-C<sub>15</sub>, for example C<sub>10</sub>-C<sub>14</sub>, alkyl group and n is at least 1, for example from 1 to 16, preferably 2 to 12, more preferably 3 to 10.

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The alkoxyated fatty alcohol nonionic surfactant will frequently have a hydrophilic-lipophilic balance (HLB)

which ranges from 3 to 17, more preferably from 6 to 15, most preferably from 10 to 15.

5 Examples of fatty alcohol ethoxylates are those made from alcohols of 12 to 15 carbon atoms and which contain about 7 moles of ethylene oxide. Such materials are commercially marketed under the trademarks Neodol 25-7 and Neodol 23-6.5 by Shell Chemical Company. Other useful Neodols include Neodol 1-5, an ethoxylated fatty alcohol averaging 11 carbon atoms in its alkyl chain with about 5  
10 moles of ethylene oxide; Neodol 23-9, an ethoxylated primary C<sub>12</sub>-C<sub>13</sub> alcohol having about 9 moles of ethylene oxide; and Neodol 91-10, an ethoxylated C<sub>9</sub>-C<sub>11</sub> primary alcohol having about 10 moles of ethylene oxide.

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Alcohol ethoxylates of this type have also been marketed by Shell Chemical Company under the Dobanol trademark. Dobanol 91-5 is an ethoxylated C<sub>9</sub>-C<sub>11</sub> fatty alcohol with an average of 5 moles ethylene oxide and Dobanol 25-7 is  
20 an ethoxylated C<sub>12</sub>-C<sub>15</sub> fatty alcohol with an average of 7 moles of ethylene oxide per mole of fatty alcohol.

Other examples of suitable ethoxylated alcohol nonionic surfactants include Tergitol 15-S-7 and Tergitol 15-S-9,  
25 both of which are linear secondary alcohol ethoxylates available from Union Carbide Corporation. Tergitol 15-S-7 is a mixed ethoxylated product of a C<sub>11</sub>-C<sub>15</sub> linear secondary alkanol with 7 moles of ethylene oxide and Tergitol 15-S-9 is the same but with 9 moles of ethylene oxide.  
30



Other suitable alcohol ethoxylated nonionic surfactants are Neodol 45-11, which is a similar ethylene oxide condensation products of a fatty alcohol having 14-15 carbon atoms and the number of ethylene oxide groups per mole being about 11. Such products are also available from Shell Chemical Company.

Further nonionic surfactants are, for example,  $C_{10}$ - $C_{18}$  alkyl polyglycosides, such as  $C_{12}$ - $C_{16}$  alkyl polyglycosides, especially the polyglucosides. These are especially useful when high foaming compositions are desired. Further surfactants are polyhydroxy fatty acid amides, such as  $C_{10}$ - $C_{18}$  N-(3-methoxypropyl) glycamides and ethylene oxide-propylene oxide block polymers of the Pluronic type.

Examples of cationic surfactants are those of the quaternary ammonium type.

The total content of surfactants in the composition is desirably 60 to 95wt%, especially 75 to 90wt%. Desirably an anionic surfactant is present in an amount of 50 to 75wt%, the nonionic surfactant is present in an amount of 5 to 50wt%, and/or the cationic surfactant is present in an amount of from 0 to 20wt%. The amounts are based on the total solids content of the composition, i.e. excluding any solvent which may be present.

The compositions, particularly when used as laundry washing or dishwashing compositions, may also independently comprise enzymes, such as protease, lipase, amylase, cellulase and peroxidase enzymes. Such enzymes are commercially available and sold, for example, under the regis-

tered trade marks Esperase, Alcalase and Savinase by No-  
vozymes. Desirably the enzymes are independently present  
in the compositions in an amount of from 0.5 to 3wt%, es-  
pecially 1 to 2wt%, when added as commercial preparations  
5 they are not pure and this represents an equivalent  
amount of 0.005 to 0.5wt% of pure enzyme.

The compositions may, if desired, independently comprise  
a thickening agent or gelling agent. Suitable thickeners  
10 are polyacrylate polymers such as those sold under the  
trade mark CARBOPOL, or the trade mark ACUSOL by Rohm and  
Haas Company. Other suitable thickeners are xanthan  
gums. The thickener, if present, is generally present in  
an amount of from 0.2 to 4wt%, especially 0.5 to 2wt%.

15 Compositions used in laundry washing / dishwashing inde-  
pendently usually comprise a detergency builder. The  
builders counteract the effects of calcium, or other ion,  
water hardness. Examples of such materials are citrate,  
20 succinate, malonate, carboxymethyl succinate, carboxy-  
late, polycarboxylate and polyacetyl carboxylate salts,  
for example with alkali metal or alkaline earth metal ca-  
tions, or the corresponding free acids. Specific exam-  
ples are sodium, potassium and lithium salts of oxydisuc-  
25 cinic acid, mellitic acid, benzene polycarboxylic acids,  
C<sub>10</sub>-C<sub>22</sub> fatty acids and citric acid. Other examples are  
organic phosphonate type sequestering agents such as  
those sold by Monsanto under the trade mark Dequest and  
alkylhydroxy phosphonates. Citrate salts and C<sub>12</sub>-C<sub>18</sub>  
30 fatty acid soaps are preferred. Further builders are;  
phosphates such as sodium, potassium or ammonium salts of  
mono-, di- or tri-poly or oligo-phosphates; zeolites;

silicates, amorphous or structured, such as sodium, potassium or ammonium salts.

Other suitable builders are polymers and copolymers known to have builder properties. For example, such materials include appropriate polyacrylic acid, polymaleic acid, and polyacrylic/polymaleic and copolymers and their salts, such as those sold by BASF under the trade mark Sokalan.

10

The builder is desirably present in an amount of up to 90wt%, preferably 15 to 90wt%, more preferable 15 to 75wt%, relative to the total weight of the composition. Further details of suitable components are given in, for example, EP-A-694,059, EP-A-518,720 and WO 99/06522.

15

The compositions can also optionally comprise one or more additional ingredients. These include conventional detergent composition components such as further surfactants, bleaches, bleach enhancing agents, builders, suds boosters or suds suppressors, anti-tarnish and anti-corrosion agents, organic solvents, co-solvents, phase stabilisers, emulsifying agents, preservatives, soil suspending agents, soil release agents, germicides, pH adjusting agents or buffers, non-builder alkalinity sources, chelating agents, clays such as smectite clays, enzyme stabilizers, anti-limescale agents, colourants, dyes, hydrotropes, dye transfer inhibiting agents, brighteners, and perfumes. If used, such optional ingredients will generally constitute no more than 10wt%, for example from 1 to 6wt%, the total weight of the compositions.

25

30

Compositions which comprise an enzyme may optionally contain materials which maintain the stability of the enzyme. Such enzyme stabilizers include, for example, polyols such as propylene glycol, boric acid and borax. Combinations of these enzyme stabilizers may also be employed. If utilised, the enzyme stabilizers generally constitute from 0.1 to 1wt% of the compositions.

The compositions may optionally comprise materials which serve as phase stabilizers and/or co-solvents. Examples are C<sub>1</sub>-C<sub>3</sub> alcohols such as methanol, ethanol and propanol. C<sub>1</sub>-C<sub>3</sub> alkanolamines such as mono-, di- and triethanolamines can also be used, by themselves or in combination with the alcohols. The phase stabilizers and/or co-solvents can, for example, constitute 0 to 1wt%, preferably 0.1 to 0.5wt%, of the composition.

The compositions may optionally comprise components which adjust or maintain the pH of the compositions at optimum levels. The pH may be from, for example, 1 to 13, such as 8 to 11 depending on the nature of the composition. For example a dishwashing composition desirably has a pH of 8 to 11, a laundry composition desirable has a pH of 7 to 9, and a water-softening composition desirably has a pH of 7 to 9. Examples of pH adjusting agents are NaOH and citric acid.

The above examples may be used for dish or fabric washing. In particular dish washing formulations are preferred which are adapted to be used in automatic dish washing machines. Due to their specific requirements

specialised formulation is required and these are illustrated below

Amounts of the ingredients can vary within wide ranges, however preferred automatic dishwashing detergent compositions herein (which typically have a 1% aqueous solution pH of above 8, more preferably from 9.5 to 12, most preferably from 9.5 to 10.5) are those wherein there is present: from 5% to 90%, preferably from 5% to 75%, of builder; from 0.1% to 40%, preferably from 0.5% to 30%, of bleaching agent; from 0.1% to 15%, preferably from 0.2% to 10%, of the surfactant system; from 0.0001% to 1%, preferably from 0.001% to 0.05%, of a metal-containing bleach catalyst; and from 0.1% to 40%, preferably from 0.1% to 20% of a water-soluble silicate. Such fully-formulated embodiments typically further comprise from 0.1% to 15% of a polymeric dispersant, from 0.01% to 10% of a chelant, and from 0.00001% to 10% of a deterative enzyme, though further additional or adjunct ingredients may be present. Detergent compositions herein in granular form typically limit water content, for example to less than 7% free water, for better storage stability.

Non-ionic surfactants useful in ADW (Automatic Dish Washing) compositions of the present invention desirably include surfactant(s) at levels of from 2% to 60% of the composition. In general, bleach-stable surfactants are preferred. Non-ionic surfactants generally are well known, being described in more detail in Kirk Othmer's Encyclopedia of Chemical Technology, 3rd Ed., Vol. 22,

pp. 360-379, "Surfactants and Detergent Systems", incorporated by reference herein.

Preferably the ADW composition comprises at least one  
5 non-ionic surfactant. One class of non-ionics are ethoxylated non-ionic surfactants prepared by the reaction of a monohydroxy alkanol or alkylphenol with 6 to 20 carbon atoms with preferably at least 12 moles particularly preferred at least 16 moles, and still more preferred at  
10 least 20 moles of ethylene oxide per mole of alcohol or alkylphenol.

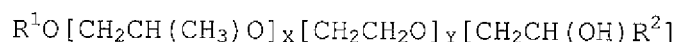
Particularly preferred non-ionic surfactants are the non-ionic from a linear chain fatty alcohol with 16-20 carbon  
15 atoms and at least 12 moles particularly preferred at least 16 and still more preferred at least 20 moles of ethylene oxide per mole of alcohol.

According to one preferred embodiment the non-ionic surfactant additionally comprise propylene oxide units in  
20 the molecule. Preferably this PO units constitute up to 25% by weight, preferably up to 20% by weight and still more preferably up to 15% by weight of the overall molecular weight of the non-ionic surfactant. Particularly  
25 preferred surfactants are ethoxylated mono-hydroxy alkanols or alkylphenols, which additionally comprises polyoxyethylene-polyoxypropylene block copolymer units. The alcohol or alkylphenol portion of such surfactants constitutes more than 30%, preferably more than 50%, more  
30 preferably more than 70% by weight of the overall molecular weight of the non-ionic surfactant.

Another class of non-ionic surfactants includes reverse block copolymers of polyoxyethylene and polyoxypropylene and block copolymers of polyoxyethylene and polyoxypropylene initiated with trimethylolpropane.

5

Another preferred non-ionic surfactant can be described by the formula:

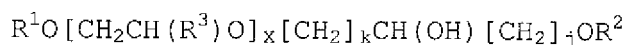


10

wherein  $R^1$  represents a linear or branched chain aliphatic hydrocarbon group with 4-18 carbon atoms or mixtures thereof,  $R^2$  represents a linear or branched chain aliphatic hydrocarbon rest with 2-26 carbon atoms or mixtures thereof,  $x$  is a value between 0.5 and 1.5 and  $y$  is a value of at least 15.

Another group of preferred nonionic surfactants are the end-capped polyoxyalkylated non-ionics of formula:

20



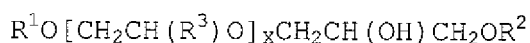
wherein  $R^1$  and  $R^2$  represent linear or branched chain, saturated or unsaturated, aliphatic or aromatic hydrocarbon groups with 1-30 carbon atoms,  $R^3$  represents a hydrogen atom or a methyl, ethyl, n-propyl, iso-propyl, n-butyl, 2-butyl or 2-methyl-2-butyl group,  $x$  is a value between 1 and 30 and,  $k$  and  $j$  are values between 1 and 12, preferably between 1 and 5. When the value of  $x$  is  $\geq 2$  each  $R^3$  in the formula above can be different.  $R^1$  and  $R^2$  are preferably linear or branched chain, saturated or unsaturated, aliphatic or aromatic hydrocarbon groups

30

with 6-22 carbon atoms, where groups with 8 to 18 carbon atoms are particularly preferred. For the group  $R^3$  H, methyl or ethyl are particularly preferred. Particularly preferred values for x are comprised between 1 and 20, preferably between 6 and 15.

As described above, in case  $x \geq 2$ , each  $R^3$  in the formula can be different. For instance, when  $x=3$ , the group  $R^3$  could be chosen to build ethylene oxide ( $R^3=H$ ) or propylene oxide ( $R^3=\text{methyl}$ ) units which can be used in every single order for instance (PO)(EO)(EO), (EO)(PO)(EO), (EO)(EO)(PO), (EO)(EO)(EO), (PO)(EO)(PO), (PO)(PO)(EO) and (PO)(PO)(PO). The value 3 for x is only an example and bigger values can be chosen whereby a higher number of variations of (EO) or (PO) units would arise.

Particularly preferred end-capped polyoxyalkylated alcohols of the above formula are those where  $k=1$  and  $j=1$  originating molecules of simplified formula:



The use of mixtures of different non-ionic surfactants is particularly preferred in ADW formulations for example mixtures of alkoxyated alcohols and hydroxy group containing alkoxyated alcohols.

The composition, such as a washing composition within the container, capsule or receptacle part, or within a compartment thereof if there is more than one compartment, need not be uniform. For example during manufacture it could be fed first with a settable agent, for example a



gel, useful in a washing process, and then with a different material. The first material could dissolve slowly in the washing process so as to deliver its charge over a long period within the washing process. This might be  
5 useful, for example, to provide delayed or sustained delivery of a softening agent in a clothes washing capsule.

The invention will now further explained with reference to the following non-limiting Figures and Examples.

10

**BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWINGS**

The foregoing summary, as well as the following detailed description of preferred embodiments of the invention,  
15 will be better understood when read in conjunction with the appended drawings. For the purpose of illustrating the invention, there are shown in the drawings embodiments which are presently preferred. It should be understood, however, that the invention is not limited to the  
20 precise arrangements shown. In the drawings:

FIG. 1 is a view from below, of a receptacle part;

FIG. 2 is a perspective view, generally from above, of  
25 the receptacle part of Fig. 1;

FIG. 3a and FIG 3b are side views along the longitudinal and equatorial axes of the receptacle part as shown in FIG.1 and FIG. 2.

30

FIGS. 1 to 3 show a receptacle part 2. The receptacle part 2 has a base wall 4. The base wall 4 is substan-

tially flat with a slight depression towards the main chamber of the receptacle part 2. The receptacle part 2 has four upright side walls 6, and has no top wall. Thus, each receptacle part 2 is upwardly open. Around  
5 its opening, at the top of the side walls 6, is an outwardly-directed flange 8, which extends around the entire opening. The flange 8 lies in one plane. When filled a cover film (not shown) may be laid over the receptacle part 2 and heat sealed against the flange 8, so that each  
10 receptacle part 2 has, over it, a closure part (not shown).

Within the main chamber defined by the base wall 4 and side walls 6 of each receptacle part 2, there is present  
15 a generally cylindrical upstand 10, in a central position. The cylindrical upstand 10 is open at its upper end, and its upper end is in the same plane as the flange 6.

20 Adjacent the cylindrical upstand 10 there is a wave-shaped upstand 12. The wave upstand 12 extends from the base wall 4 of the receptacle part to the side walls 6 on the longitudinal axis, up to the height of the side walls 6.

25 The cylindrical upstand 10 and the wave upstand 12 divide the main chamber of the receptacle part into three separate compartments. There is a first compartment (A) within the volume defined by the cylindrical upstand 10.  
30 There are two further compartments created by the dividing effect of the wave upstand 12; a second compartment (B) on the cylindrical upstand side 10 of the wave up-

stand, being above the first compartment and a third compartment (C) on the opposite side of the wave upstand 12. For clarity these three compartments are shown with the letter references (A), (B) and (C) in FIG 2. The cover  
5 film is preferably sealed against the wave upstand 12, thus dividing the receptacle into separate compartments as described above.

### CONTAINER MANUFACTURING PROCESS

10

This process describes a method of manufacturing a container as illustrated in the accompanying FIGS. 1-3. In the filling stage it is filled with the compositions as set out in the Composition Examples.

15

#### **The Moulding Stage**

Containers according to the invention were made by the injection moulding method. The injection cavities were  
20 in a two-impression (cap/body) composite water-cooled stainless-steel mould. The PVOH had a material melt flow index of 10-20 grams/10 min (DIN 53735). Injection temperatures were 175°C, 180°C, 180°C and 185°C in the feed, zone 2 and 3, and Nozzle areas. The first stage injection  
25 pressure was 400 psi, and the hold stage pressure was 270 psi. The pressure well time was 3 seconds in the first stage and 5 seconds in the hold stage. Tool temperatures were between ambient and 40°C.

30 The moulding pressures were just sufficient to fill the cavities on the first pressure stage and then sufficient packing pressure to hold on the second stage. Mould open

and close rates were as fast as possible.

The moulds were such that an array of container receptacle parts were moulded simultaneously. In this case up  
5 to 64 receptacle parts could be moulded in an array. Where an array was formed separation of the receptacle parts was necessary (see later).

Usually each receptacle weighed 2.0 to 2.5g. The resin  
10 used for moulding comprised 85% PVOH, 11% Sorbitol and 4% processing aids.

#### **The Filling Stage**

15 In the preferred embodiment of the invention where the container is a multi-chamber product as illustrated in the accompanying FIGS. a distinct detergent composition was added to each of the compartments. This allowed the formation of a multi-function detergent container which  
20 could be used to achieve each of the required functions in, for example, an automatic dishwasher. Indeed the following example is in the field of automatic dishwashing.

25 For compartment (A) generally a solidified melt formulation was added. This formulation typically comprised a surfactant which could be liquefied at elevated temperature, such as 60°C, (see Composition Examples). This formulation was added at elevated temperature in the molten state using a hot-melt nozzle dispensing device. After  
30 addition of the rinse aid the receptacle parts were placed in a cooling system to allow the rinse aid to so-

lidify. The amount of this formulation added was usually 0.4 g.

For compartment (B) generally a powder formulation was added. This formulation was added using a powder nozzle dispensing device. For this stage of addition, vibration of the receptacle part to aid settling of the powder was employed. Also a compression station was used to aid levelling. The amount of this formulation added was usually 0.4 g.

For compartment (C) generally a gel formulation was added. This formulation typically comprised a plurality of materials suspended in a thickened gel formulation. The formulation could be liquefied at elevated temperature, such as 60°C, (see Composition Examples). This formulation was added at elevated temperature in the molten state using a hot-melt nozzle dispensing device. The amount of this formulation added was usually 6.4 g.

#### **The Sealing Stage**

The filled receptacle parts were passed by a wetting station, comprising a "wet plate" made of sand blasted aluminium.

A sealing top film (PVOH, 80-100µm) was then applied to the wet receptacle parts.

The film was pressed onto the receptacle parts using a flat sealing surface which has been modified to follow the seal contour along the flange of the receptacle part

and the wave upstand. The sealing surface operated at 150°C and pressed for 1.5 seconds.

#### **The Separation Stage**

5

A laser or other IR source was arranged to focus on the area of the join between individual receptacle parts in the array. The laser was applied to cut the joins thus freeing up the individual containers.

10

#### **Further Steps**

Once sealed the containers have to be perforated on the powder compartment top film to allow vapours to exhaust  
15 (e.g. those vapours arising from decomposition of the bleach components of the powder (where present) during storage). This was done with a plate with needles.

The containers were then removed from the trays to be  
20 packed.

COMPOSITION EXAMPLES

Composition Example 1: Phosphorus Containing Composition

Raw Material	COMPARTMENT A  Rinse Aid (0.4 g)	COMPARTMENT B  Powder (10.4g)	COMPARTMENT C  Gel (6.4 g)
Sodium tripolyphosphate		43	
Sodium carbonate		16	
Sodium percarbonate		22	
Phosphate speckles		4	
Benzotriazol		0.4	
HEDP 4 Na (88,5%)		0.3	
Protease <sup>1</sup>		1.5	
Amylase <sup>1</sup>		1	
TAED		6	
1,2-Propylenediglycol		1	
Dye		0.02	
Perfume		0.1	
Sulphonated Polymer <sup>2</sup>		5	
Sulphonated Polymer <sup>2</sup>			5
Surfactant <sup>3</sup>			24
Polyglykol <sup>4</sup>			9
1,2-Propylendiglycol			1
Dye			0.03
Antifoam <sup>5</sup>			0.25
TAED			3
Sodium tripolyphosphate			57.5
Polyglycol 6000			0.3
Surfactant <sup>6</sup>	100		
	100	100	100

5

1 Granules which contain approx. 10% active enzyme.

2 AMPS co-polymer.

3 Non-ionic low foaming surfactant.

4 Mixed poly alkoxylate.

10 5 Silicone oil.

6 Non-ionic surfactant (m.pt. approx. 50°C)

The receptacle part weighed 2.5g and comprised 85% of low molecular weight PVOH (degree of hydrolysis 85-88%), 11%

sorbitol and 4% processing aids. The receptacle lid comprised a PVOH foil (90 $\mu$ m thick).

5 The contents of Compartment A (the non-ionic surfactant (6)) were heated at 55°C until molten. The contents were added into Compartment A and chilled to solid. The powder was filled into Compartment B. The gel was heated to 65°C and stirred for 20 minutes. The gel was filled into Compartment C and allowed to chill.

10

The receptacles were then sealed with PVOH film.



Composition Example 2: Phosphorus-Reduced Composition

Raw Material	COMPARTMENT A  Rinse Aid (0.4 g)	COMPARTMENT B  Powder (10.4g)	COMPARTMENT C  Gel (6.4 g)
Sodium Citrate		26	
Sodium tripolyphosphate		19	
Sodium carbonate		16	
Sodium percarbonate		22	
Phosphate speckles		4	
Benzotriazol		0.4	
HEDP 4 Na (88,5%)		0.35	
Protease <sup>1</sup>		1.5	
Amylase <sup>1</sup>		1	
TAED		6	
1,2-Propylenediglycol		1	
Dye		0.02	
Perfume		0.1	
Sulphonated Polymer <sup>2</sup>		5	
Sulphonated Polymer <sup>2</sup>			5
Surfactant <sup>3</sup>			24
Polyglykol <sup>4</sup>			9
1,2-Propylendiglycol			1
Dye			0.03
Antifoam <sup>5</sup>			0.25
TAED			3
Sodium tripolyphosphate			57.5
Polyglycol 6000			0.3
Surfactant <sup>6</sup>	100		
	100	100	100

1 Granules which contain approx. 10% active enzyme.

5 2 AMPS co-polymer.

3 Non-ionic low foaming surfactant.

4 Mixed poly alkoxylate.

5 Silicone oil.

6 Non-ionic surfactant (m.pt. approx. 50°C)

10

The preparation was as for Example 1.

Composition Example 3: Phosphorus Free Composition

Raw Material	COMPARTMENT A  Rinse Aid (0.4 g)	COMPARTMENT B  Powder (10.4g)	COMPARTMENT C  Gel (6.4 g)
Tri-sodium citrate		51.5	
Sodium carbonate		15	
Sodium percarbonate		20	
Benzotriazol		0.4	
HEDP 4 Na (88,5%)		0.3	
Protease <sup>1</sup>		1.5	
Amylase <sup>1</sup>		1	
1,2-Propylenediglycol		1	
Dye		0.02	
Perfume		0.1	
Acrylate homo polymer		3	
Sulphonated Polymer <sup>2</sup>			5
Surfactant <sup>3</sup>			24
Polyglykol <sup>4</sup>			9
1,2-Propylendiglycol			1
Dye			0.03
Antifoam <sup>5</sup>			0.25
TAED			8
Tri-sodium citrate			51.75
Polyglycol 35000			1
Surfactant <sup>6</sup>	100		
	100	100	100

1 Granules which contain approx. 10% active enzyme.

5 2 AMPS co-polymer.

3 Non-ionic low foaming surfactant.

4 Mixed poly alkoxyate.

5 Silicone oil.

6 Non-ionic surfactant (m.pt. approx. 50°C)

10

The preparation was as for Example 1.

Composition Example 4: Phosphorus Containing Composition  
with a bleach activator composition in the centred compartment

Raw Material	COMPARTMENT A  Melt (1.5 g)	COMPARTMENT B  Powder (9.2g)	COMPARTMENT C  Gel (6.4 g)
Sodium tripolyphosphate		52	
Sodium carbonate		15	
Sodium percarbonate		20	
Phosphate speckles		4	
Benzotriazol		0.4	
HEDP 4 Na (88,5%)		0.3	
Protease <sup>1</sup>		1.5	
Amylase <sup>1</sup>		1	
1,2-Propylenediglycol		1	
Dye		0.02	
Perfume		0.1	
Sulphonated Polymer <sup>2</sup>		5	
Sulphonated Polymer <sup>2</sup>			5
Surfactant <sup>3</sup>			24
Polyglykol <sup>4</sup>			9
1,2-Propylendiglycol			1
Dye			0.03
Antifoam <sup>5</sup>			0.25
Sodium tripolyphosphate			61
Polyglycol 6000			0.3
Gelatine	3		
Glycerine	47		
TAED	50		
	100	100	100

5

1 Granules which contain approx. 10% active enzyme.

2 AMPS co-polymer.

3 Non-ionic low foaming surfactant

4 Mixed poly alkoxylate grade.

10 5 Silicone oil

The preparation was as for Example 1.

Composition Example 5: Phosphate Containing Composition  
Including PAP

Raw Material	COMPARTMENT A  (1.5 g)	COMPARTMENT B  Powder (9.2 g)	COMPARTMENT C  Gel (6.4 g)
Sodium tripolyphosphate		42.5	
Sodium carbonate		16	
Tri-sodium citrate		22	
Phosphate speckles		4	
Benzotriazol		0.4	
HEDP 4 Na (88,5%)		0.3	
Protease <sup>1</sup>		1.5	
Amylase <sup>1</sup>		1	
TAED		6	
1,2-Propylenediglycol		1	
Dye		0.02	
Perfume		0.1	
Sulphonated Polymer <sup>2</sup>		5	
Sulphonated Polymer <sup>2</sup>			5
Surfactant <sup>3</sup>			24
Polyglykol <sup>4</sup>			9
1,2-Propylendiglycol			1
Dye			0.03
Antifoam <sup>5</sup>			0.25
TAED			3
Sodium tripolyphosphate			57.5
Polyglycol 6000			0.3
PAP <sup>6</sup>	100		
	100	100	100

- 5    1 Granules which contain approx. 10% active enzyme.  
       2 AMPS co-polymer.  
       3 Non-ionic low foaming surfactant.  
       4 Mixed poly alkoxylate grade.  
       5 Silicone oil  
 10   6 PAP granules of particle size > 250µm.

In the container used in this Example the cylindrical part is raised to the height of the side wall. The PAP is added as a powder. The remainder of the preparation process is as for Example 1.

5

### Rigidity Examples

#### **Rigidity Example 1**

10

A receptacle part as illustrated in the accompanying Figures was made in an injection moulding process. The receptacle part had the overall dimensions of 40mm (length), 28mm (width) and 18mm (height). The thickness of the receptacle was 300 $\mu$ m with a rim thickness of 700 $\mu$ m.

15

The composition of the receptacle part was the same as that of the composition Examples.

20

The rigidity of the receptacle part was measured using a measuring device available from Imatec in accordance with the DIN 878 method. Using this apparatus a force of 0.8N was applied to the receptacle part (across its width) with a needle having a width of 2.4mm.

25

Measurements were taken immediately after production of the receptacle part and after storage in moist conditions (after 15 minutes and 30 minutes at 30°C and 70% humidity). The rigidity data, shown in terms of the deformation extent of the receptacle part is shown in the following table.

30

The following table also contains values for the brittleness of the receptacle part, in terms of the force required to break the container, measured using the same equipment.

5

Receptacle	Deformation (mm)			Brittleness (N)
	Minimum	Average	Maximum	
Immediately After Production	0.54	0.89	1.14	<80
15 min. at 30°C / 70% humidity	1.10	1.33	1.69	-
15 min. at 30°C / 70% humidity	1.38	1.69	2.76	>100

10 The deformation ability of the receptacle party increases significantly after plasticisation by water in the humid atmosphere. This has the beneficial effect that as the receptacle part has a low deformation ability (high rigidity) after production it can be filled and sealed effectively without any incorrect filling / poor sealing. After plasticisation the container has a high deformation  
15 ability (low rigidity) which is pleasing to a consumer on a tactile level.

The container also has a much lower brittleness after plasticisation. This allows the container to be transported and handled without causing rupture of the container.  
20